# PATENT COOPERATION TREATY

# PCT

REC'D 30 SEP 2005

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## INTERNATIONAL PRELIMINARY REPORT ON PATENTABILITY

(Chapter II of the Patent Cooperation Treaty)

(PCT Article 36 and Rule 70)

| Applicant's or agent's file reference<br>ACD 3008 WO | **FOR FURTHER ACTION** See Form PCT/IPEA/416 | |
|---|---|---|
| International application No.<br>PCT/EP2004/006601 | International filing date *(day/month/year)*<br>18.06.2004 | Priority date *(day/month/year)*<br>27.06.2003 |
| International Patent Classification (IPC) or national classification and IPC<br>C08F4/00, C08F2/22 | | |
| Applicant<br>AKZO NOBEL N.V. | | |

1. This report is the international preliminary examination report, established by this International Preliminary Examining Authority under Article 35 and transmitted to the applicant according to Article 36.
2. This REPORT consists of a total of 7 sheets, including this cover sheet.
3. This report is also accompanied by ANNEXES, comprising:
   a. ☒ *sent to the applicant and to the International Bureau)* a total of 3 sheets, as follows:
      ☐ sheets of the description, claims and/or drawings which have been amended and are the basis of this report and/or sheets containing rectifications authorized by this Authority (see Rule 70.16 and Section 607 of the Administrative Instructions).
      ☐ sheets which supersede earlier sheets, but which this Authority considers contain an amendment that goes beyond the disclosure in the international application as filed, as indicated in item 4 of Box No. I and the Supplemental Box.
   b. ☐ *(sent to the International Bureau only)* a total of (indicate type and number of electronic carrier(s)) , containing a sequence listing and/or tables related thereto, in computer readable form only, as indicated in the Supplemental Box Relating to Sequence Listing (see Section 802 of the Administrative Instructions).

4. This report contains indications relating to the following items:

| | | |
|---|---|---|
| ☒ | Box No. I | Basis of the opinion |
| ☐ | Box No. II | Priority |
| ☐ | Box No. III | Non-establishment of opinion with regard to novelty, inventive step and industrial applicability |
| ☐ | Box No. IV | Lack of unity of invention |
| ☒ | Box No. V | Reasoned statement under Article 35(2) with regard to novelty, inventive step or industrial applicability; citations and explanations supporting such statement |
| ☐ | Box No. VI | Certain documents cited |
| ☐ | Box No. VII | Certain defects in the international application |
| ☒ | Box No. VIII | Certain observations on the international application |

| Date of submission of the demand | Date of completion of this report |
|---|---|
| 24.12.2004 | 29.09.2005 |
| Name and mailing address of the international preliminary examining authority:<br>European Patent Office - P.B. 5818 Patentlaan 2<br>NL-2280 HV Rijswijk - Pays Bas<br>Tel. +31 70 340 - 2040 Tx: 31 651 epo nl<br>Fax: +31 70 340 - 3016 | Authorized Officer<br>Droghetti, A<br>Telephone No. +31 70 340-4143 |

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# INTERNATIONAL PRELIMINARY REPORT ON PATENTABILITY

International application No.
PCT/EP2004/006601

## Box No. I Basis of the report

1. With regard to the **language**, this report is based on the international application in the language in which it was filed, unless otherwise indicated under this item.

   ☐ This report is based on translations from the original language into the following language , which is the language of a translation furnished for the purposes of:
   - ☐ international search (under Rules 12.3 and 23.1(b))
   - ☐ publication of the international application (under Rule 12.4)
   - ☐ international preliminary examination (under Rules 55.2 and/or 55.3)

2. With regard to the **elements*** of the international application, this report is based on *(replacement sheets which have been furnished to the receiving Office in response to an invitation under Article 14 are referred to in this report as "originally filed" and are not annexed to this report)*:

**Description, Pages**

| | |
|---|---|
| 2-26 | as originally filed |
| 1 | received on 24.12.2004 with letter of 23.12.2004 |

**Claims, Numbers**

| | |
|---|---|
| 5-14 | as originally filed |
| 1-4 | received on 24.12.2004 with letter of 23.12.2004 |

☐ a sequence listing and/or any related table(s) - see Supplemental Box Relating to Sequence Listing

3. ☐ The amendments have resulted in the cancellation of:
   - ☐ the description, pages
   - ☐ the claims, Nos.
   - ☐ the drawings, sheets/figs
   - ☐ the sequence listing *(specify)*:
   - ☐ any table(s) related to sequence listing *(specify)*:

4. ☐ This report has been established as if (some of) the amendments annexed to this report and listed below had not been made, since they have been considered to go beyond the disclosure as filed, as indicated in the Supplemental Box (Rule 70.2(c)).
   - ☐ the description, pages
   - ☐ the claims, Nos.
   - ☐ the drawings, sheets/figs
   - ☐ the sequence listing *(specify)*:
   - ☐ any table(s) related to sequence listing *(specify)*:

* *If item 4 applies, some or all of these sheets may be marked "superseded."*

**Box No. V Reasoned statement under Article 35(2) with regard to novelty, inventive step or industrial applicability; citations and explanations supporting such statement**

1. Statement

| | | | |
|---|---|---|---|
| Novelty (N) | Yes: | Claims | 1-14 |
| | No: | Claims | |
| Inventive step (IS) | Yes: | Claims | |
| | No: | Claims | 1-14 |
| Industrial applicability (IA) | Yes: | Claims | 1-14 |
| | No: | Claims | |

2. Citations and explanations (Rule 70.7):

**see separate sheet**

**Box No. VIII Certain observations on the international application**

The following observations on the clarity of the claims, description, and drawings or on the question whether the claims are fully supported by the description, are made:

**see separate sheet**

**Re Item V**

Reference is made to the following documents:

D1: US A 5155192 (cited by the applicant)
D2: US A 2002/0123591 ("")
D3: US A 3778422
D4: US B 6399728

**1.** The amendments introduced with letter dated 23.12.2004 are allowable in view of Article 34(2)(b) PCT.

**2. Novelty (Art. 33(2) PCT)**

The subject-matter of claims 1-14 is novel in view of D1-D4. The reasons as follows.

**2.1.** D1 discloses a (co)polymerization process wherein an organic peroxide (e.g. the same peroxydicarbonate) and an hydroperoxide used as stabilizer are involved.
Being the same peroxydicarbonate, also the water solubility of the organic peroxide initiator has to be the same as claimed (see search report). The same consideration applies to the half-life of the peroxydicarbonate in D1.
Note that the hydroperoxide is used to stabilize the initiator composition and that this composition is used in a polymerization process in an amount such that at the start of the polymerisation the peroxydicarbonate (initiator) is present at 0.01-3 wt%, calculated on the monomer. However there is no hint in D1 that the peroxide is added to the polymerisation mixture during the process and at the polymerisation temperature as claimed (see col. 5, lines 41-52).
The subject-matter of claims 1-14 is therefore not novel in view of D1.

**2.2.** D2 describes a polymerisation process wherein organic peroxide initiator as claimed are used.
The organic peroxide can be introduced at the polymerisation temperature are as claimed (see search report).
However no specific reference to the controlling agents (e.g. hydroperoxides) as claimed is present in D2.

Thus the subjectmatter of claims 1-14 is novel in view of D2.

**2.3.** D3 discloses a (co)polymerization process wherein an organic peroxide (e.g. the same peroxydicarbonate) and an hydroperoxide as claimed (stabilizer) are involved.
However there is no hint in D3 that the peroxide is added to the polymerisation mixture during the process and at the polymerisation temperature as claimed.
Thus the subject-matter of claims 1-14 is novel in view of D3.

**2.4.** In D4 a stabilized composition of organic peroxydicarbonate and a maleate stabilzer as claimed is described.
No hint to the addition of the organic peroxide at the polymerisation temperature during the polym. process.
Therefore the subject-matter of claims 1-14 is novel in view of D4.

**3. Inventive Step (Art. 33(3) PCT)**

The subject-matter of claims 1-14 does not involve an inventive step in the sense of Article 56 EPC for the following reasons.

D2, which is regarded as the closest prior art, discloses a polymerization process wherein an organic peroxide (e.g. diacyl peroxide, see examples, tables II and VII) is used as initiator in a polymerisation process. Note that in D2 the initiator has the same half-life as claimed and it is added at the polymerization temperature ("hot-start" as claimed). In this case the use of a polymerization inibitor-stabilizer (a radical trapping compound) together with the initiator is preferred (see page 2, parag. 17 in D2; as in claim 9 and in the description on page 3, lines 1-9 of the present application).
Polymers with reduced fish eyes levels are obtained.

The present application differs from D2 in that specific stabilizers or controlling agents (e.g. hydroperoxides) are used together with the organic peroxide initiators.
According to the examples on file the use of the distinguishing feasture above leads polymers with a further reduced level of fish-eye defects over D2.
Thus the problem to be solved by the present invention may therefore be regarded as the provision of

polymerisation process for preparing polymers with an improved (that is reduced) level of fish eye defects. The solution proposed in claims 1-14 of the present application cannot be considered as involving an inventive step (Article 33(3) PCT) for the following reasons.

The use of a controlling agents as claimed for stabilizing the peroxide initiators is well-known in the art (see in D1, D3 or D4).
In particular, D3 (see col. 2, lines 4-14) discloses a polymerisation process carried out in the presence of organic peroxides as claimed (e.g. peroxydicarbonates) and the same hydroperoxides, in order to obtain a more uniform polymer in molecular weight and molecular structure, and as a consequence (as exaplained by the applicant on page 1-2, lines 23-2) with a reduced level of fish eyes.

Note that the use of maleate stabilizer for peroxide initiators is also well known in the art (see D4, search report).

Thus the skilled man starting from D2 and trying to provide an process for preparing polymers with a impoved reduced fish eye level, would have found in D3 (and D1, D4) a hint to use controllinh agents as claimed in order to solve the problem posed.

Thus the subject-matter of claims 1-14 cannot be regarded as inventive in view of D1-D4.

**3.** The subject-matter of claims 1-14 meets the requirements of **Article 33(4) PCT**, with regard to **industrial applicability**.

**Re Item VIII (Art. 6 PCT)**

**1.** In claim 1 the matter for which protection is sought is not defined. The claim attempts to define the subject-matter in terms of the result to be achieved (see the expresssion "effective amount" referring to the controlling agent). Such a definition is only allowable under the conditions elaborated in the Guidelines C-III, 4.7. In this instance, however, such a formulation is not allowable because it appears possible to define the subject-

matter in more concrete terms, viz. in terms of how the effect is to be achieved (see pages 8-9, lines 22-7). The same applies to claims 13-14.

**2.** The applicant is reminded that the sentences starting with the term "preferably" do not limit the scope of the claims. This applies in claims 1,7,11.

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EPO - DG 1

24. 12. 2004

(87)

# POLYMERIZATION PROCESS FOR PREPARING (CO)POLYMERS

The present invention relates to an aqueous dispersion polymerization process for preparing a (co)polymer wherein one or more organic peroxides are used as initiator (as a source of free radicals) in conjunction with an effective amount of one or more controlling agents. The invention also relates to formulations comprising organic peroxide(s) and an effective amount of said controlling agent(s) suitable for use in said aqueous dispersion polymerization process. The invention finally relates to (co)polymers obtainable by the dispersion polymerization process.

Over the years, there has been a large number of publications describing the polymerization of ethylenically unsaturated monomers using an organic peroxide as initiator. For example, US 5,155,192 discloses storageable and/or transportable compositions containing peroxydicarbonate to which an organic peroxide has been added to retard the decomposition of said peroxydicarbonate. The compositions of US 5,155,192 are suitable for use in the conventional mass, suspension, or emulsion (co)polymerization of ethylenically unsaturated monomers. In US 5,155,192 no further specifications of the peroxydicarbonates to be used are given, such as their solubility or their half life.

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An unwanted side effect frequently observed in conventional polymerization processes is the formation of so-called fish eyes in the (co)polymer. One explanation for fish eyes is that they are caused by small quantities of polymer material having a molecular weight that differs considerably from the average molecular weight of the rest of the polymer material. Due to a difference in melt property between said polymer material and the "average" polymer material, irregularities can occur in the final shaped polymer material. It will be clear that this phenomenon is undesirable, for example, for the transparency and uniformity of the final (co)polymer material, particularly in thin films. Furthermore, the presence of fish eyes may even be detrimental to the strength

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A

US 3,778,422 pertains to a process for the production of vinyl halide polymers in which vinyl halide monomers are polymerized in the presence of an initiator that comprises an organic peroxydicarbonate.

US 6,399,728 describes a process for the polymerization of vinyl chloride using a thermally stabilized initiator composition comprising at least one dialkyl peroxydicarbonate and a stabilizing effective amount of a compound of the general structure R-O-C(=O)-CH=CH-C(=O)-O-R.

Amended claims

1. Polymerization process for preparing a (co)polymer wherein one or more organic peroxides selected from the group consisting of diacyl peroxides, peroxyesters, peroxydicarbonates, and mixtures thereof are used in conjunction with an effective amount of one or more controlling agents selected from the group consisting of organic hydroperoxides, ethylenically unsaturated organic compounds that preferably cannot homopolymerize, compounds with labile carbon-hydrogen bonds, oximes, and mixtures thereof, with the proviso that the solubility of the peroxydicarbonate(s) in water at 0°C is at least 5 ppm, preferably the solubility of all organic peroxides in water at 0°C is at least 5 ppm, and wherein the process is a conventional aqueous dispersion polymerization process or an aqueous dispersion polymerization process wherein at least part of the one or more organic peroxides used as initiator is dosed to the reaction mixture at the polymerization temperature.

2. A polymerization process according to claim 1 wherein the one or more organic peroxides are selected from the group of diacyl peroxides, peroxyesters, and mixtures thereof

3. A polymerization process according to claim 2 wherein the one or more organic peroxides have a solubility in water at 0°C of at least 5 ppm

4. A polymerization process according to any one of claims 1 to 3 wherein the one or more organic peroxides are selected from the group consisting of organic peroxides having a half-life of at least 0.0001 hour and at most 1.0 hour at the polymerization temperature and mixtures thereof

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